# FACTORS RELATED TO THE FLAVOR STABILITY OF FOAM-DRIED WHOLE MILK. I. EFFECT OF OXYGEN LEVEL[1]

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SUMMARY

After developing methods capable of packaging milk powders in atmospheres of nitrogen containing less than 0.1% oxygen, a study was made of the effect of the in-package oxygen levels on the storage life of foam-dried whole milk. It was found that the shelf life of this product could be extended by reducing the oxygen levels in the packages below those now generally considered acceptable for the storage of spray-dried whole milk powders. During an 8-mo. period, 14 foam-dried milks were made from fluid milks, each of which received a minimum heat treatment of 145° F. for 30 min. The dry milks contained 2-3% moisture, were packaged in an atmosphere that contained about 0.1, 1, and 21% oxygen, and stored at 80° F.; they were flavor-scored each 2 mo. for 6 mo. by a ten-judge expert panel, using a slight modification of the American Dairy Science Association scoring guide. A statistically, highly significant improvement in storage stability was noted in milk powders packed in 0.1% oxygen compared with those packed in 1% oxygen and in air. Numerical trends in oxidized and other off-flavors are reported.

A new method for drying whole milk has been devised recently in the laboratories of this Division (11). This foam-dried powder can be easily reconstituted to a milk of beverage quality. The powder as initially prepared by a batch process is bulky and subject to off-flavor development during storage. Research was undertaken to overcome these defects. This paper reports results obtained during a study of the effect of in-package oxygen levels on the storage life of foam-dried whole milk powders.

The effect of in-package oxygen levels on the shelf life of spray-dried whole milk has been the subject of numerous investigations. Although there has been a general consensus of opinion among investigators that reduced levels of oxygen in the package retard the development of oxidized flavor in whole milk powders, no agreement exists as to the minimum levels needed during storage. Lea *et al.* (9) concluded that the development of oxidized flavors in spray-dried whole milk could be controlled by reducing the oxygen content in the free space of the container to 1 to 3%. Coulter (4) and Coulter *et al.* (5) concluded that less than 1% oxygen in the packing gas was necessary to prevent oxidation of the whole milk powder. Schaffer (10) reported 3% as the upper limit of in-package oxygen content for extended storage life at room temperature. In the light of this information, and in view of the fact that nothing was known about stabilizing the flavor of foam-dried milk, methods for packaging products in nitrogen containing controlled amounts of oxygen were developed and a study

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of the effect of in-package oxygen levels on the storage life of foam-dried whole milk was undertaken.

## METHODS

*Preparation of foam-dried whole milk.* The original procedure (13) for the production of foam-dried milk was employed with the following exceptions: (a) The concentrate was heated to 145° F. and homogenized, at 4,000 and 500 p.s.i. pressure on the first and second stages, respectively; (b) nitrogen was metered into the concentrate through a capillary tube and dispersed by passing the 110° F. mixture twice through a two-stage homogenizer, using 500 p.s.i. pressure on both stages. After drying, the moisture content of the comminuted foams varied between 2.0 and 3.2% before packaging. Moisture, dispersibility, and free fat were determined as described previously (13). Bulk density was determined by tamping 10 g. of dry milk in a 100-ml. graduate cylinder to the minimum volume and dividing ten by the minimum volume in milliliters.

*Packaging.* Dry milks were packed within one day after preparation in 208 by 208 tin cans, ⅔ of the cans (Series 1 and 2) were sealed with lids in which 1/32-in. holes drilled into a patch of solder were left open, and the rest of the cans (Series 3) were covered with lids but not sealed. The samples were placed in a vacuum shelf drier, Series 1 and 2 on circular discs which could be rotated, permitting every can to appear in front of a window containing two ⅝-in. holes closed with rubber stoppers. After holding the cans overnight below 1-mm. pressure at room temperature, the vacuum was broken with nitrogen (Seaford grade—oxygen content below 0.1%) to a slight excess over atmospheric pressure. The stoppers in the window were removed and a soldering iron and a rod for turning the disc were inserted. Series 1 was sealed while excess pressure was maintained continually in the drier. Series 2 was prepared by again drawing a vacuum to below 1-mm. pressure, breaking with nitrogen containing 1% oxygen, and sealing while using the same precautions. The vacuum was drawn once more, broken with air, and Series 3 sealed with a can sealer. The cans were tested for leaks by placing them under deaerated water in a desiccator and pulling a vacuum. Samples were discarded if bubbles escaped from seams or seals. Packing under three levels of oxygen: below 0.1%, at 1%, and in air for all batches of dry milk was accomplished by this procedure. The oxygen content of the interstitial gas in the containers was determined by the Haldane method (7) in two samples of the low oxygen packs, drawn from duplicate cans without oxygen contamination. Using the packing procedure described, it was found that the lowest level of oxygen packs always contained below 0.1% oxygen. The intermediate oxygen level packs ranged from 1.1 to 1.3% oxygen. The moisture content of the dry milk in the cans was reduced to approximately 2% because of the overnight evacuation. After packaging, the cans were stored at 80° F. After initial tasting, the packaged, stored dry milks were periodically tasted and scored at ages up to six months, by ten selected judges.

## TASTE PANEL PROCEDURES

*Selection of judges.* Prior to this study, flavor thresholds of potential judges were evaluated for specific flavors, so that a statistically homogeneous taste panel could be developed. Since cooked and oxidized flavors were expected to predominate in this and projected studies, they were the ones utilized in screening the judges. Various intensities of the off-flavors in milk were prepared by dilution. This diluting technique was successfully used by Kirkpatrick (8) and others (1-3, 6). The samples of the various concentrations thus prepared were presented to the judges in randomized pairs. The judges were asked to determine which sample of each pair possessed more off-flavor. The judges choosing the greatest number of samples correctly were used for the panel. These judges were later proven to be homogeneous, since they had similar judgment of a particular sample. This group of judges, which was used throughout the entire flavor study, should be considered qualified, because the likes and dislikes of flavor were not important as they would be for a consumer panel.

*Testing experimental milks.* After the group selection was completed, ten judges met almost daily in a temperature-controlled taste-panel room ($70 \pm 2°$ F.) where they tasted and scored about ten samples at each sitting.

In each experiment, the dry milks were first tasted within one or two days after preparation (initial) and at intervals after storage (usually after 2, 4, and 6 mo.). They were reconstituted (12.6% T.S.) with the proper proportion of chlorine-free spring water during the afternoon preceding the tasting period, which took place at 11 o'clock on the following morning. Samples were kept in a refrigerator overnight and warmed to 95° F. the next day. The milk cooled to 75-80° F. in the cups before tasting. The judges independently evaluated the flavor of the samples and recorded a numerical score corresponding to the flavors they found in each sample. The scoring guide used in these studies was similar to the one adopted by the American Dairy Science Association and is shown in Table 1.

*Evaluation of the scores.* The initial score, or the score after 2, 4, or 6 mo. of storage at 80° F. for a particular sample, was represented by the average

TABLE 1

Flavor score guide used throughout these studies

| Criticisms | Slight | Definite | Pronounced |
|---|---|---|---|
| Acid | 36-38 | 33-35 | 29-32 |
| Astringent | 37-39 | 33-36 | 28-32 |
| Bitter | 34-37 | 29-33 | 25-28 |
| Chalky | 37-39 | 33-36 | 29-32 |
| Cooked | 38-40 | 35-37 | 32-34 |
| Feed | 38-40 | 35-37 | 32-34 |
| Flat | 38-40 | 36-37 | 34-35 |
| Foreign, objectionable | 20-25 | 10-19 | 0-9 |
| Foreign, unobjectionable | 36-38 | 33-35 | 30-32 |
| Metallic | 32-34 | 28-31 | 24-27 |
| Oily (greasy) | 34-35 | 32-33 | 30-31 |
| Oxidized | 33-36 | 29-32 | 24-28 |
| Rancid (lipolysis) | 20-25 | 10-19 | 0-9 |
| Salty | 36-38 | 33-35 | 30-32 |
| Scorched | 34-36 | 31-33 | 28-30 |
| Stale | 34-36 | 31-33 | 28-30 |
| Unclean | 34-36 | 31-33 | 28-30 |
| Weedy | 32-34 | 28-31 | 24-27 |

score of the ten judges. The flavor data obtained by the ten taste-panel judges were analyzed for significance by the F-test or t-test (12).

## RESULTS AND DISCUSSION

*The effect of oxygen level in the package on dry milk flavor.* The effects of the three levels of oxygen on the flavor score average during 6 mo. at 80° F. are presented in Figure 1. Fourteen dry milks were prepared by a standard procedure for eight months. Storage stability increased with decrease in oxygen content, which indicates that oxidative processes are causes for deterioration of foam-dried whole milk during storage. The initial average score, 35.6, decreased to 35 in about 1 wk. when the milks were kept in air, in about 3 wk. if kept at 1% oxygen, and after 4½ mo. at 0.1% oxygen. A great improvement in storage stability resulted from packing at 0.1% as compared to 1% oxygen, the level obtained with commercial nitrogen pack. Results from the literature (4, 5, 9, 10) indicate that a higher level of oxygen can be tolerated in spray-dried whole milk packages, which may be due to the fact that it is less bulky and consequently each can has less gas available for the fat. Also, spray-dried milk may have received more heat treatment. The effect of heat treatment on the storage stability of foam-dried whole milk will be the subject of a subsequent paper. The initial scores of the powders varied between 35 and 37, with one exception. No relationship was apparent between scores and bulk density, free fat, dispersibility, or moisture.

Data from the flavor tests, as evaluated by statistical treatment, for the effect of oxygen level in the package are summarized in Table 2. The results of the t-test showed that the samples with 1% oxygen were significantly better than those packaged in air; likewise, samples packed in 0.1% oxygen scored higher than those packaged with 1% oxygen. Statistical analysis of the data permits prediction of the range in flavor scores which can be expected with different oxygen contents in the canned samples after storage.

TABLE 2

A comparison of the flavor scores of foam-dried milks containing various levels of oxygen after storage for 6 mo. at 80° F.

| Oxygen levels compared | Standard error of average flavor score difference | t-Value required for significance at 0.1% level | Degrees of freedom | Probability level at which significance occurred | t-Value calculated | Confidence limits for average flavor score difference** |
|---|---|---|---|---|---|---|
| | | | *(n − 1)* | *(%)* | | *(95% confidence level)* |
| 0.1 vs. 1 | 0.214 | 4.22 | 13 | 0.1 | 4.86 | 1.04 ± 0.43 |
| 1 vs. air | 0.438 | 4.22 | 13 | 0.1 | 4.78 | 2.09 ± 0.88 |
| 0.1 vs. initial | 0.210 | 4.22 | 12* | 1.0 | 3.94 | 0.83 ± 0.42 |
| 1 vs. initial | 0.270 | 4.22 | 12* | 0.1 | 6.63 | 1.70 ± 0.54 |
| Air vs. initial | 0.288 | 4.22 | 12* | 0.1 | 13.33 | 3.84 ± 0.57 |

* Data on one was excluded because its initial score was exceptionally low.

** The confidence limits indicate the range within which the true difference in flavor scores for the compared oxygen levels would be expected to fall: The average plus and minus two times the standard error.

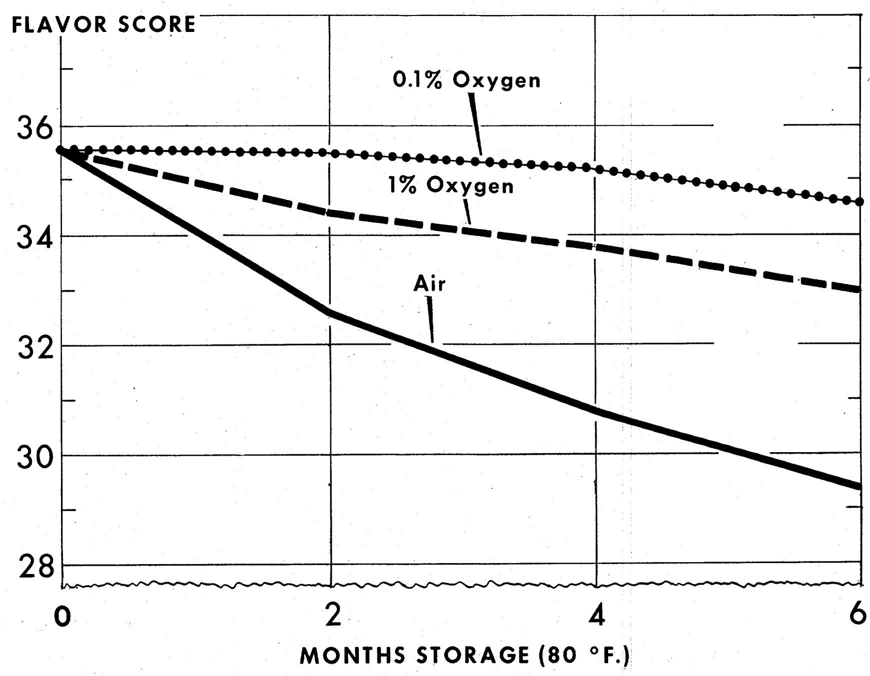


FIG. 1. Flavor score of foam-dried whole milk during storage. Averages of 14 samples.

A comparison of the flavor score means of dry milks packed at various levels of oxygen, initially and after 6 mo. storage at 80° F., is shown in Table 2. This indicates the decrease of the flavor score from its initial value that would be expected after 6 mo. storage. Data on one dry milk with an exceptionally low initial score was not included. This indicates that the samples packaged at the 0.1% oxygen level and stored for 6 mo. at 80° F. did not differ significantly from their initial values. There was a greater decrease in flavor score in the 1% oxygen packed samples similarly stored for the same period. The air packed samples were the poorest and they had the greatest decrease in flavor score. These differences were all statistically analyzed at the 0.1% level of significance. A score range difference at 0.4 to 1.2; 1.3 to 2.3; and 3.3 to 4.4 was found between initial and 6-mo.-old samples held at 80° F. and that were packaged in 0.1, 1, and 21% oxygen (air) atmospheres, respectively.

Table 3 is a compilation of the most frequent off-flavors reported by the judges when tasting milks reconstituted after storage at 80° F. in atmospheres of 0.1% and 1% oxygen, and in air. The oxidized flavor was predominant at all levels of oxygen. Trends in this and other flavors during 6 mo. of storage are noteworthy.


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TABLE 3

A distribution of frequencies of various predominating off-flavors as influenced by oxygen level and period of storage at 80° F.

| Oxygen level | Storage 80° F. | Flavor criticism (per cent occurrence) | | | | | | |
|---|---|---|---|---|---|---|---|---|
| | | Astrin-gent | Cooked | Feed | Oxi-dized | Stale | Un-clean | Others |
| *(%)* | *(mo.)* | | | | | | | |
| Initial | 0 | 13[a] | 27[a] | 3[a] | 21[a] | 13[a] | 5[a] | 18[a] |
| 0.1 | 2 | 14 | 24 | 2 | 36 | 6 | 7 | 11 |
| | 4 | 12 | 19 | 2 | 35 | 12 | 7 | 13 |
| | 6 | 7 | 13 | 1 | 46 | 11 | 7 | 15 |
| 1 | 2 | 10 | 14 | 1 | 56 | 3 | 8 | 8 |
| | 4 | 8 | 9 | 1 | 52 | 11 | 4 | 15 |
| | 6 | 5 | 7 | 1 | 64 | 12 | 3 | 8 |
| Air | 2 | 8 | 12 | 2 | 60 | 7 | 3 | 8 |
| | 4 | 4 | 5 | 1 | 70 | 9 | 3 | 8 |
| | 6 | 2 | 7 | 1 | 75 | 8 | 2 | 5 |

[a] Only one sample was tested initially for all oxygen levels.

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